

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 2 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

1. A method of preparing a carbon-doped group III-V compound semiconductor crystal, comprising the steps of:

placing a compound raw material, solid carbon, and a boron oxide substance into a crucible or a boat,

sealing said crucible or boat containing said compound raw material, said solid carbon, and said boron oxide substance within an airtight vessel formed of a gas impermeable material,

heating and melting said compound raw material in said crucible or said boat sealed within said airtight vessel, and

solidifying said melted compound raw material to grow a carbon-doped compound semiconductor crystal,

wherein an amount of said solid carbon placed into said crucible or said boat is larger than an amount of carbon doped into said compound semiconductor crystal.

2. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising a step of heating and melting said boron oxide substance and having said melted boron oxide substance in contact with at least a portion of said solid carbon, during said step of heating and melting said compound raw material.

3. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said gas impermeable material comprises a material selected from the group consisting of quartz and pBN.

Applicant : Tomohiro Kawase et al
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 3 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

4. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said boron oxide substance comprises boron oxide and water.

5. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 4, wherein said boron oxide substance contains 10-500 wt ppm of said water.

6. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said amount of said solid carbon placed into said crucible or said boat is at least 10 times larger than said amount of carbon doped into said compound semiconductor crystal.

7. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising a step of subjecting said solid carbon to a heat treatment under reduced pressure before placing said solid carbon into said crucible or said boat.

8. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 7, comprising carrying out said heat treatment for 1 hour to 12 hours at a temperature of 500° C. – 2000° C. under a pressure of 1 Torr – 1×10^{-8} Torr.

9. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material to grow said crystal.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 4 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

10. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 9, wherein said step of maintaining said melted compound raw material in a melted state is carried out for 3-72 hours.

11. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said solid carbon comprises powder carbon.

12. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 11, wherein said powder carbon has a grain size of not more than 100 μm .

13. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said solid carbon comprises fiber carbon.

14. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 13, wherein said fiber carbon has an average diameter of not more than 50 μm .

15. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said solid carbon comprises bulk carbon.

16. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 15, wherein said bulk carbon has a disk shape with a disk diameter smaller than an inner diameter of said crucible.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 5 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

17. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 15, wherein said bulk carbon comprises a sintered compact of carbon powder.

18. A method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said crucible or said boat comprises pBN.

19. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, wherein said compound raw material comprises GaAs, and wherein said compound semiconductor crystal comprises a GaAs crystal.

20. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 2, further comprising having said melted boron oxide substance in contact with at least a portion of said melted compound raw material, during said step of heating and melting said compound raw material.

21. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, further comprising selecting a target amount of said carbon to be doped into said compound semiconductor crystal, and adjusting said amount of said solid carbon placed into said crucible or said boat so as to responsively achieve said target amount of said carbon to be doped into said semiconductor crystal.

22. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 1, carried out such that said carbon-doped compound semiconductor crystal has a variation of carbon concentration of not more than 8 1/3% between a lowest carbon concentration and a highest carbon concentration, relative to said lowest carbon concentration.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 6 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

Cancel claims 23-25.

26. Vertical boat growth of single crystal, semi-insulating GaAs ingots having controlled planned target levels of Carbon therein comprising: (a) loading a crucible with a charge of poly-crystal GaAs material; a source of carbon; and Boron Oxide over a selectively oriented seed crystal; (b) placing said crucible in a closed quartz tube; (c) applying a controlled pattern of heating to melt the charge and a portion of the seed crystal to sequentially freeze the melt starting at the interface with the seed crystal to form a single crystal; wherein said source of carbon is carbon powder in a selected quantity having a defined large nominal doping potential compared to the planned target level of Carbon in an as grown ingot; and said Boron Oxide is provided in an amount for providing spacer material between an as grown ingot and a crucible wall, and between a seed crystal and the bottom of said crucible.

27. Vertical boat growth of single crystal, semi-insulating GaAs ingots in accordance with claim 26 wherein said pattern of heating comprises: heating said charge to the melting point temperature of GaAs; holding that temperature for a period of time.

28. Vertical boat growth of single crystal, semi-insulating GaAs ingots in accordance with claim 26 wherein the nominal doping potential of said carbon powder included in the charge is the order of 100 times the planned target level of carbon dopant in an as grown ingot.

29. Vertical boat growth of single crystal, semi-insulating GaAs ingots in accordance with claim 26 wherein the nominal doping potential of said carbon powder included in the charge is at least several times the planned target level of carbon dopant in an as grown ingot.

30. Cancel claim 30 without prejudice.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 7 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/IT)

31. The method of any of claims 1 - 22 wherein sufficient boron oxide substance is placed in said crucible or boat so that the boron oxide substance surrounds the melted semiconductor compound.

32. The method of claim 31 wherein said melting and solidifying is conducted in a vertical furnace.

33. The method of any of claims 1 - 22 wherein said melting and solidifying is conducted in a vertical furnace.

34. The method of any of claims 2 - 10 or 18 - 22 wherein said solid carbon is powdered carbon.

35. The method of claim 34 wherein sufficient boron oxide substance is placed in said crucible or boat so that the boron oxide substance surrounds the melted semiconductor compound.

36. The method of claim 34 wherein said melting and solidifying is conducted in a vertical furnace.

37. The method of any of claims 2 - 10 or 18 - 22 wherein said solid carbon is carbon fibers.

38. The method of claim 37 wherein sufficient boron oxide substance is placed in said crucible or boat so that the boron oxide substance surrounds the melted semiconductor compound.

39. The method of claim 37 wherein said melting and solidifying is conducted in a vertical furnace.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 8 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

40. (Amended) A method of preparing a carbon-doped group III-V compound semiconductor comprising the steps of:

melting a boron oxide substance in contact with carbon, thereby forming a boron oxide - carbon mixture,

heating and melting a III-V compound semiconductor raw material together with said boron oxide - carbon mixture in a boat,

maintaining said compound raw material in melted form in said boat for a period to permit carbon to migrate from said boron oxide - carbon mixture into said compound raw material, and

solidifying said melted compound raw material in said boat to form a crystalline carbon-doped compound semiconductor,

wherein the amount of carbon in the initial boron oxide - carbon mixture is larger than the amount of carbon doped into said compound semiconductor.

41. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 40, wherein said boron oxide substance comprises boron oxide and water.

42. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 41, wherein said boron oxide substance contains 10-500 wt ppm of said water.

43. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 40, wherein said amount of said carbon in contact with said melted boron oxide substance is at least 10 times larger than said amount of carbon doped into said crystalline semiconductor.

44. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 40, further comprising a step of subjecting solid carbon

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 9 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

to a heat treatment under reduced pressure before melting said boron oxide substance in contact with said carbon.

45. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 44, comprising carrying out said heat treatment for 1 hour to 12 hours at a temperature of 500° C. - 2000° C. under a pressure of 1 Torr - 1x 10⁻⁸ Torr.

46. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 45, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material.

47. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 46, wherein said step of maintaining said melted compound raw material in a melted state is carried out for 3 - 72 hours.

48. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 40, further comprising selecting a target amount of said carbon to be doped into said compound semiconductor crystal, and adjusting said amount of said carbon in contact with said melted boron oxide substance so as to responsively achieve said target amount of said carbon to be doped into said semiconductor crystal.

49. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 40, wherein said carbon comprises powder carbon.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 10 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

50. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 42, wherein said carbon comprises powder carbon.

51. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 40, wherein said carbon comprises fiber carbon.

52. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 42, wherein said carbon comprises fiber carbon.

53. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 40, wherein said compound raw material comprises GaAs, and wherein said compound semiconductor crystal comprises a single crystal of GaAs.

54. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, carried out such that said carbon-doped compound semiconductor crystal has a variation of carbon concentration of not more than 8-1/3% between a lowest carbon concentration and a highest carbon concentration, relative to said lowest carbon concentration.

55. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, wherein said boron oxide substance comprises boron oxide and water.

56. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 55, wherein said boron oxide substance contains 10-500 wt ppm of said water.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 11 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

57. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, wherein said amount of said carbon in contact with said melted boron oxide substance is at least 10 times larger than said amount of carbon doped into said compound semiconductor crystal.

58. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, further comprising a step of subjecting solid carbon to a heat treatment under reduced pressure before melting said boron oxide substance in contact with said carbon.

59. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 58, comprising carrying out said heat treatment for 1 hour to 12 hours at a temperature of 500° C. - 2000° C. under a pressure of 1 Torr - 1×10^{-8} Torr.

60. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material to grow said crystal.

61. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 60, wherein said step of maintaining said melted compound raw material in a melted state is carried out for 3-72 hours.

62. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, further comprising selecting a target amount of said carbon to be doped into said compound semiconductor crystal, and adjusting said amount of said carbon in contact with said melted boron oxide substance so as to responsively achieve said target amount of said carbon to be doped into said semiconductor crystal.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 12 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

63. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, wherein said carbon comprises powder carbon.

64. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 56, wherein said carbon comprises powder carbon.

65. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 53, wherein said carbon comprises fiber carbon.

66. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 56, wherein said carbon comprises fiber carbon.

67. The method of preparing a carbon-doped group III-V compound semiconductor according to claim 40, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material.

68. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 43, wherein said carbon comprises powder carbon.

69. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 43, wherein said carbon comprises fiber carbon.

70. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 57, wherein said carbon comprises powder carbon.

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 13 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

71. The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 57, wherein said carbon comprises fiber carbon.

72. (New) A method of preparing a carbon-doped group III-V compound semiconductor comprising the steps of:

melting a boron oxide substance in contact with carbon, thereby forming a boron oxide - carbon mixture,

heating and melting a III-V compound semiconductor raw material together with said boron oxide - carbon mixture,

maintaining said compound raw material in melted form for a period to permit carbon to migrate from said boron oxide - carbon mixture into said compound raw material, and

solidifying said melted compound raw material to form a crystalline carbon-doped compound semiconductor,

wherein the amount of carbon in the initial boron oxide - carbon mixture is larger than the amount of carbon doped into said compound semiconductor,

further comprising a step of subjecting said carbon to a heat treatment under reduced pressure before melting said boron oxide substance in contact with said carbon.

73. (New) The method of preparing a carbon-doped group III-V compound semiconductor according to claim 72, comprising carrying out said heat treatment for 1 hour to 12 hours at a temperature of 500° C. - 2000° C. under a pressure of 1 Torr - 1x 10⁻⁸ Torr.

74. (New) The method of preparing a carbon-doped group III-V compound semiconductor according to claim 73, further comprising a step of maintaining said melted compound raw material in a melted state for a certain time period before said step of solidifying said melted raw material.

Applicant : Tomohiro Kawase et al
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 14 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

75. (New) The method of preparing a carbon-doped group III-V compound semiconductor according to claim 74, wherein said step of maintaining said melted compound raw material in a melted state is carried out for 3 - 72 hours.

76. (New) A method of preparing a carbon-doped group III-V compound semiconductor comprising the steps of:

melting a boron oxide substance in contact with fiber carbon, thereby forming a boron oxide - carbon mixture,

heating and melting a III-V compound semiconductor raw material together with said boron oxide - carbon mixture,

maintaining said compound raw material in melted form for a period to permit carbon to migrate from said boron oxide - carbon mixture into said compound raw material, and

solidifying said melted compound raw material to form a crystalline carbon-doped compound semiconductor,

wherein the amount of carbon in the initial boron oxide - carbon mixture is larger than the amount of carbon doped into said compound semiconductor.

77. (New) The method of preparing a carbon-doped group III-V compound semiconductor according to claim 76, wherein said boron oxide substance contains 10-500 wt ppm of said water.

78. (New) The method of preparing a carbon-doped group III-V compound semiconductor according to claim 76, wherein said amount of said fiber carbon in contact with said melted boron oxide substance is at least 10 times larger than said amount of carbon doped into said crystalline semiconductor.

79. (New) The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 76, wherein said compound raw material

Applicant : Tomohiro Kawase et al.
Serial No. : 09/824,965
Filed : April 3, 2001
Page : 15 of 17

Attorney's Docket No.: 12967-002001 / 997047-06
(TM/it)

comprises GaAs, and wherein said compound semiconductor crystal comprises a single crystal of GaAs.

80. (New) The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 79, wherein said boron oxide substance contains 10-500 wt ppm of said water.

81. (New) The method of preparing a carbon-doped group III-V compound semiconductor crystal according to claim 79, wherein said amount of said fiber carbon in contact with said melted boron oxide substance is at least 10 times larger than said amount of carbon doped into said compound semiconductor crystal.